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THERMAL CONDUCTIVITY MEASUREMENTS OF A CANDIDATE VIKING HEAT-SHIELD MATERIAL AFTER STERILIZATION, AND DURING EXPOSURE TO VACUUM, AND TO A SIMULATED MARTIAN ATMOSPHERE

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REFERENCE: Greenwood, Lawrence R. and Fleming, Roy M., "Thermal Conductivity Measurements of a Candidate Viking Heat-Shield Material After Sterilization, and During Exposure to Vacuum, and to a Simulated Martian Atmosphere," ASTM/IES/AIAA Space Simulation Conference, September 14-16, 1970.

ABSTRACT: An experimental program has been conducted to measure the thermal conductivity of the proposed Viking heat-shield material, Martin SLA-561, after sterilization (60 hours at 276° F) and during exposure to vacuum and to a simulated Martian atmosphere (74.4 percent CO₂, 12.8 percent N₂, 12.8 percent Ar). In situ thermal conductivity measurements were made at 75° F using the line-source technique. The thermal conductivity of SLA-561 was measured to be 0.0298 Btu/ft-hr-°F at atmospheric pressure. In the first environmental sequence consisting of atmosphere, vacuum, and simulated Martian atmosphere exposure, a 60-percent reduction in thermal conductivity was measured in vacuum. After a 4-hour exposure to a 7-torr pressure in the simulated Martian atmosphere, the thermal conductivity increased 67 percent from the value measured in vacuum. The second environmental sequence consisted of measurements in the atmosphere, after sterilization and during exposure to vacuum and the simulated Martian atmosphere. The results of these measurements showed that sterilization had no effect on the thermal conductivity measured at atmospheric pressure nor on the changes measured in vacuum and in the simulated Martian atmosphere. Thermal conductivity was measured at varying pressures during both environmental sequences

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and the results showed the thermal conductivity to be essentially independent of pressure below 10^{-4} torr with the transition from molecular to continuum flow occurring in the 10^{-2} to 10 torr pressure range.

KEY WORDS: thermal conductivity, vacuum effect, ablation material

Recent experimental results have shown that the thermal and mechanical properties of spacecraft materials can be altered by exposure to the space environment.^{1,2,3} In particular, results have shown that the thermal and mechanical properties of ablative heat-shield materials may change due to sterilization and exposure to vacuum. To determine the thermal conductivity of a candidate Viking heat-shield material during the mission environmental sequence, an experimental program has been conducted wherein in situ thermal conductivity measurements were made after sterilization and during exposure to vacuum and a simulated Martian atmosphere.

EXPERIMENTAL PROGRAM

Thermal Conductivity Measurements

The line-source technique was used for measuring thermal conductivity.¹ In this technique a heater wire and thermocouple wire are placed inside a sample as shown in Fig. 1. Heat is generated in the sample by passing a known heater current through the heater wire, and the variation of temperature with time is measured with the thermocouple located on the heater wire. The heater circuit consisted of a 6-volt battery, an ammeter, and a variable resistor for presetting the heater current prior to initiating sample heating. After initiating the heater current, the temperature-time history of the heater wire is recorded using the thermocouple located on the heater wire, a reference junction and a recorder. From the recorder output, a computer program is used to fit a least-squares straight line to the data and calculate the thermal conductivity from the slope of the line. The thermal conductivity was calculated from⁴

$$K = \frac{q}{4\pi(\Theta_2 - \Theta_1)} \ln \frac{t_2}{t_1}$$

where

K = thermal conductivity
q = heat input

Θ = temperature

t = time after initiation of heat generation

Fig. 2 is a photograph of the thermal conductivity instrumentation and Fig. 3 is a photograph showing details of the sample instrumentation. All measurements were made at 200 mA heater current with a heating period of 5 minutes. A second thermocouple was inserted 1/4 inch (see Fig. 3) below the sample surface and monitored during the tests to determine if the surface temperature was changing. Also, a plot of the logarithm time-temperature output was also examined to check the linearity. All measurements reported are the average of the thermal conductivity measured on three samples at a given time.

Environmental Chamber

The sterilization, and exposure to vacuum, and a simulated Martian atmosphere was done in the 20-cu-ft-ultrahigh vacuum chamber shown in Fig. 2. This is a stainless steel chamber capable of achieving pressures of 10^{-10} torr. A complete description of the chamber is given in Ref. 5.

Material

The candidate Viking heat-shield material studied was SLA-561, made by the Martin Marietta Corporation. The SLA-561 has a density of 0.201 g/cc and is composed of a silicone elastomer filled with silica spheres, phenolic microballoons, silica fibers, and cork. A complete description of the material, including its ablative properties, can be found in Ref. 6. Thermal conductivity samples were 4 by 4 by 6 inches, and typical samples are shown in Fig. 4. The 0.010-inch manganin heater wires and the 30-gage copper-constantan thermocouple wires were cast into the heat-shield samples during sample preparation. The material was preconditioned several weeks at 45 percent relative humidity in a class 100 clean room prior to testing.

Environmental Sequence

Thermal conductivity measurements were made during two environmental sequences: (1) atmosphere, vacuum, and simulated Martian atmosphere exposure, and (2) atmosphere, sterilization, vacuum, and simulated Martian atmosphere exposure.

The first sequence began by making several thermal conductivity measurements on the three samples in the vacuum chamber at atmospheric pressure (see Fig. 3). The chamber was then evacuated and thermal conductivity measurements were made as a function of time during the 128.5-hour vacuum exposure time. After 146.1 hours, the pressure was increased to 7.0 torr with a simulated Martian atmosphere consisting of 74.4 percent CO_2 , 12.8 percent N_2 , and 12.8 percent Ar. Thermal conductivity

measurements were made at 7.0 torr. The chamber pressure was then reduced to 1.0, 0.5, and 0.1 torr and thermal conductivity measurements were made at each of these pressures. The pressure time history for sequence 1 is shown in Fig. 5. All environmental exposures and thermal conductivity measurements were at 75° F.

At the start of the second sequence, three different thermal conductivity samples were sterilized in the chamber by heating the samples of 276° F for 60 hours at a pressure of 600 torr in a dry nitrogen atmosphere. After sterilization the thermal conductivity was measured at 75° F and the vacuum exposure begun. Thermal conductivity measurements were made as a function of vacuum exposure time for 95.0 hours and after 96.5 hours the pressure was raised to 7.0 torr with the simulated Martian atmosphere. The pressure time history for this sequence is also shown in Fig. 5.

EXPERIMENTAL RESULTS

Sequence 1 - Atmosphere, Vacuum, Simulated Martian Atmosphere

The thermal conductivity measured at atmospheric pressure was 0.0298 Btu/ft-hr-°F as shown on Fig. 6. After 1.8 hours in vacuum, the average thermal conductivity had been reduced 46 percent to 0.0161 Btu/ft-hr-°F. For the first 20 hours in vacuum the thermal conductivity continued to decrease, and after 31.5 hours the conductivity was 0.0120 Btu/ft-hr-°F and remained approximately constant for the remainder of the vacuum exposure period. After 146.1 hours the pressure in the chamber was raised to 7 torr with the simulated Martian atmosphere. Measurements were made immediately after reaching 7 torr and after 4, 8, and 12 hours. The thermal conductivity measured at these times was 0.0186, 0.0200, 0.0191, and 0.0193, respectively. The pressure was then reduced to 1.0 torr for 4 hours, after which time the thermal conductivity was 0.0145 Btu/ft-hr-°F. The pressure was then reduced to 0.5 torr for 4 hours after which time the thermal conductivity was 0.0140 Btu/ft-hr-°F. Finally, the pressure was reduced to 0.1 torr for 4 hours after which time the thermal conductivity was 0.0131 Btu/ft-hr-°F.

Sequence 2 - Sterilization, Vacuum, Simulated Martian Atmosphere

A second environmental sequence on three different SLA-561 samples was conducted wherein thermal sterilization was included. The thermal conductivity measured at 600 torr in a nitrogen atmosphere after sterilization was 0.0296 Btu/ft-hr-°F (see Fig. 7). Since the thermal conductivity for the unsterilized samples discussed in sequence 1 was 0.0298 at atmospheric pressure, there was essentially no effect of sterilization on the thermal conductivity. After sterilization the vacuum

exposure began, and the measured thermal conductivity values are shown in Fig. 7. The vacuum-induced changes for the sterilized samples (sequence 2) were similar to the vacuum-induced changes for the unsterilized samples (sequence 1). For instance, after 2 hours in vacuum, the thermal conductivity of the sterilized samples (sequence 2) was 0.0161, whereas the thermal conductivity of the unsterilized samples (sequence 1) was 0.0155 Btu/ft-hr-°F. After 95 hours in vacuum, the thermal conductivity of the sterilized samples (sequence 2) was 0.0140 Btu/ft-hr-°F compared with 0.0124 Btu/ft-hr-°F for the unsterilized samples (sequence 1).

After 96.5 hours the pressure was increased to 7.0 torr with a simulated Martian atmosphere. Three hours later the thermal conductivity for the sterilized samples (sequence 2) was 0.0212 Btu/ft-hr-°F. The thermal conductivity for the unsterilized samples (sequence 1) was 0.0200 Btu/ft-hr-°F after 4 hours in a 7-torr atmosphere of the simulated Martian environment. The sterilization had very little effect on the thermal conductivity of SLA-561 during vacuum exposure and exposure to the simulated Martian atmosphere at 7 torr.

Effects of Pressure

The effect of pressure for both the unsterilized (sequence 1) and sterilized (sequence 2) samples is shown in Fig. 8. First, it should be noted that there are no discernible differences in the thermal conductivity measured at the same pressure for either of the sequences studied. The measurements do indicate that at pressures below 10^{-4} torr the thermal conductivity may be independent of pressure. In the pressure region 10^{-2} to 10 torr, the characteristic knee in the thermal conductivity curve (due to transition from molecular to continuum flow) was observed. At about 10^{-1} torr, there appear to be differences in the thermal conductivity measured during the vacuum chamber pumpdown and those measured in the simulated Martian atmosphere. This is difficult to confirm because the vacuum pumpdown measurements were made while the pressure was changing as a function of time (see Fig. 5) and thus it cannot be assumed that the samples were allowed sufficient time to establish equilibrium with their environment.

CONCLUSIONS

The results of making in situ thermal conductivity measurements on SLA-561 during sequence 1 (atmosphere, vacuum, simulated Martian atmosphere) and sequence 2 (atmosphere, sterilization, vacuum, simulated Martian atmosphere) resulted in the following conclusions:

1. The thermal conductivity of the unsterilized samples (sequence 1) was reduced by 46 percent after 1.8 hours in vacuum and by 60 percent after 31.5 hours. The thermal

conductivity after 12 hours in a 7-torr pressure of a simulated Martian atmosphere was 0.0193 Btu/ft-hr-°F. The thermal conductivity did not appear to be a function of time in vacuum after 31.5 hours of exposure.

2. There was essentially no effect of sterilization on either the thermal conductivity measured at atmospheric pressure or on the vacuum-induced changes in thermal conductivity. Measurements at 7 torr for both sterilized and unsterilized samples were approximately the same.

3. The thermal conductivity measured for both environmental sequences was essentially independent of pressure below 10^{-4} torr. In the 10^{-2} to 10 pressure region the thermal conductivity was a function of pressure.

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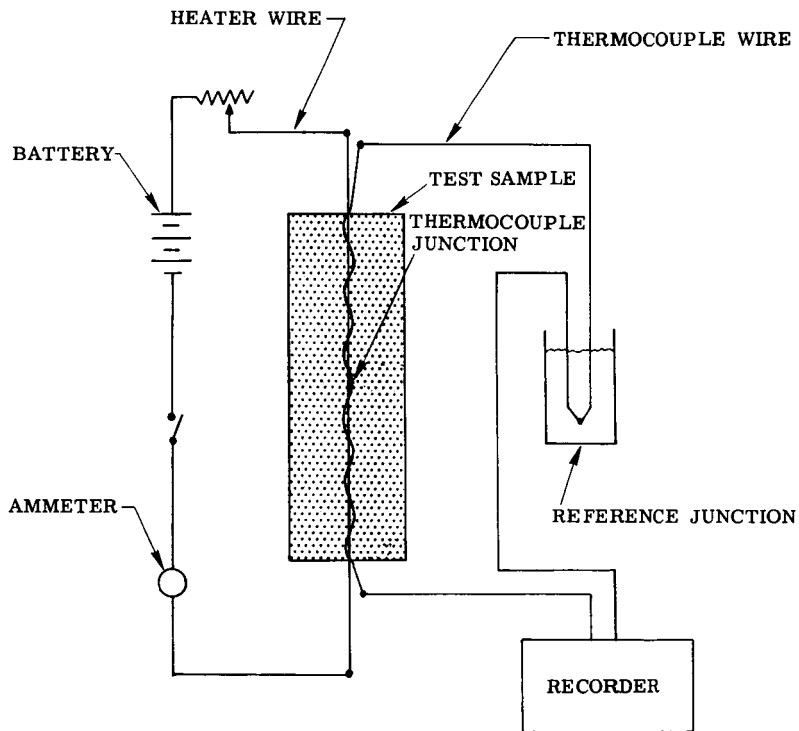


Figure 1.- Experimental technique used for measuring thermal conductivity.

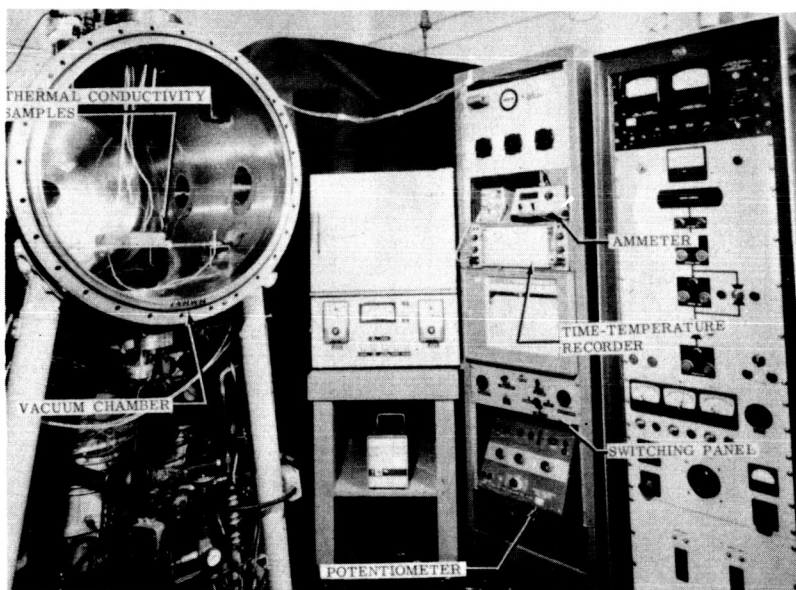


Figure 2.- Experimental apparatus used in making the thermal conductivity measurements.

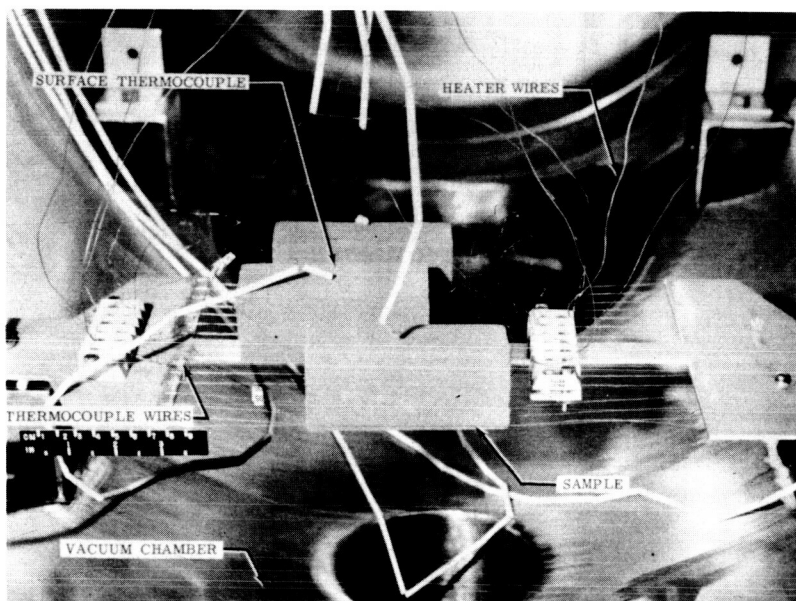


Figure 3.- Heat-shield samples located in the 20-cubic-foot ultrahigh vacuum chamber.

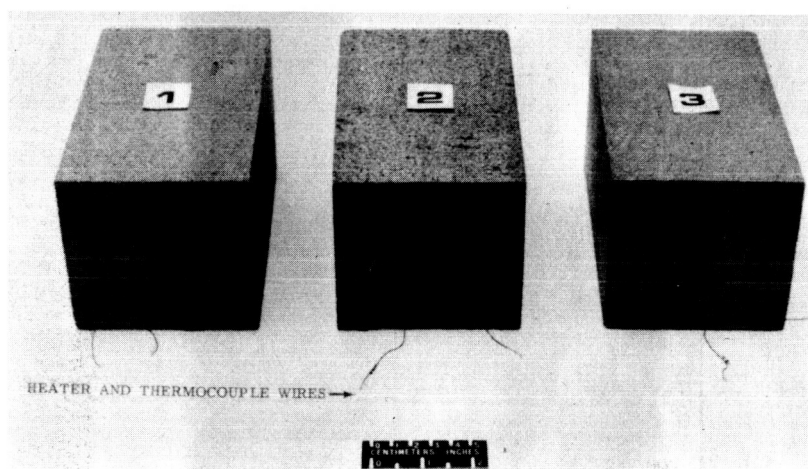


Figure 4.- Heat-shield samples used in the thermal conductivity study.

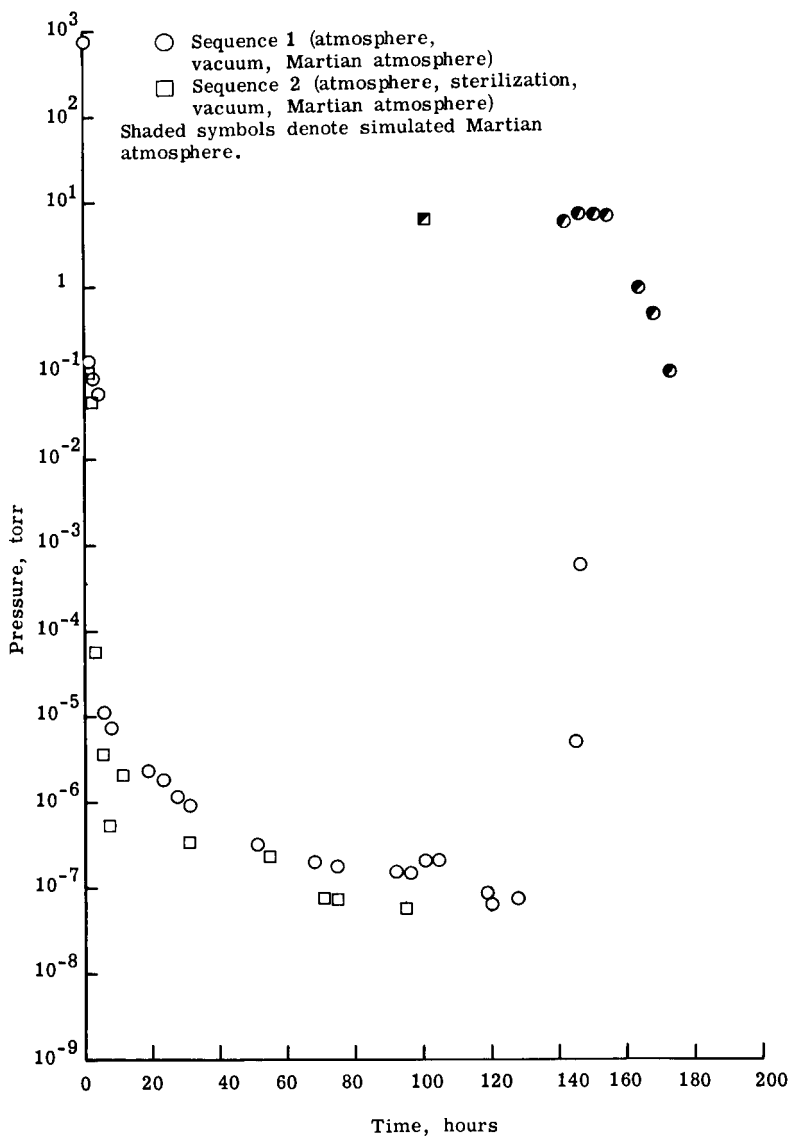


Figure 5.- Pressure time history during thermal conductivity study.

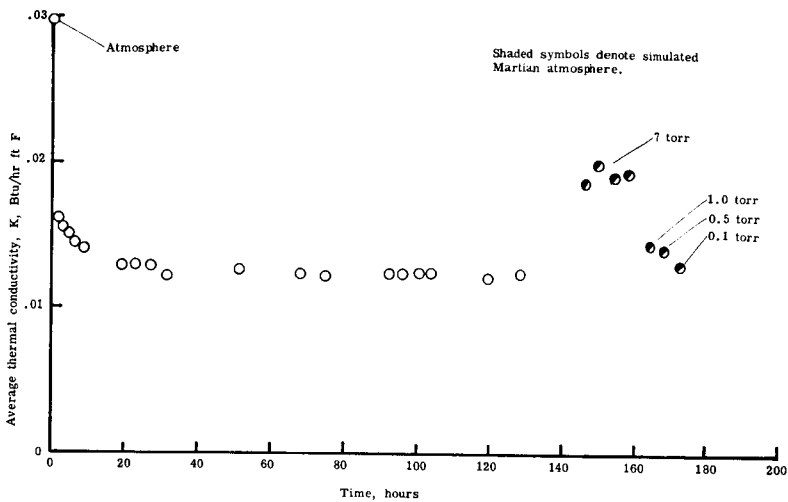


Figure 6.- Thermal conductivity of SLA-561 measured for sequence 1 (atmosphere, vacuum, Martian atmosphere).

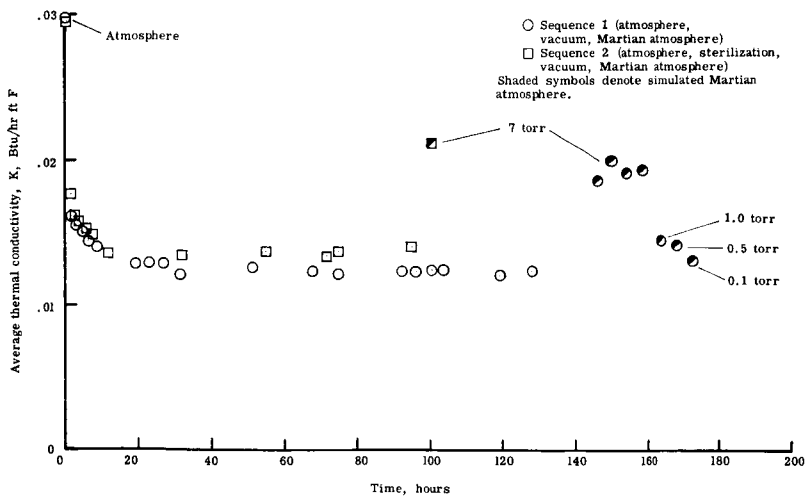


Figure 7.- Thermal conductivity of SLA-561 measured for sequence 1 and sequence 2.

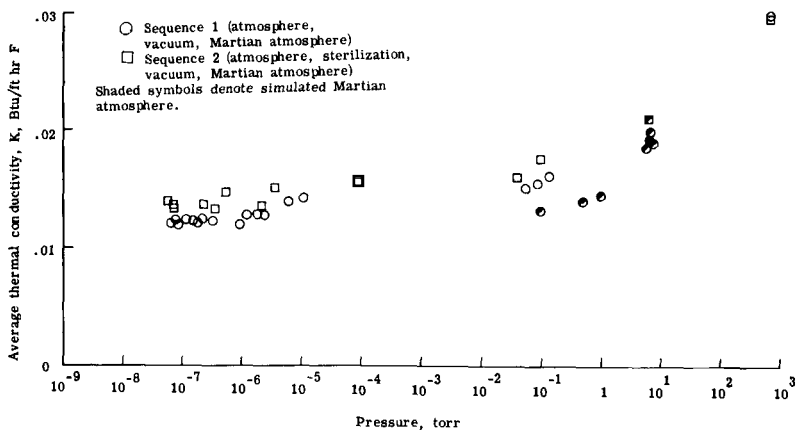


Figure 8.- Effect of pressure on the thermal conductivity of SLA-561.